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पोटैशियम कार्बोनेट,  
अनहाइड्रोस — विशिष्टि  
( दूसरा पुनरीक्षण )

Potassium Carbonate,  
Anhydrous — Specification  
( Second Revision )

ICS 71.060

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## FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by the Chemical Division Council.

This standard was originally published in 1973 and was first revised in 1992. In the first revision, a new requirement for Sodium along with the relevant test method was incorporated. An alternate test method for Chloride determination was included, while some modifications were made in other test methods also. In this second revision, alternate test methods for determination of Lead have been incorporated. Further, Induced Coupled Plasma Optical Emission Spectroscopy (ICP-OES) method for the determination of Iron, Lead, Calcium, Magnesium and Sodium has also been added.

Potassium carbonate is used as a dehydrating agent and for making other potassium salts, such as chlorates and chromates. It also finds its use in ceramics, explosives, fertilizers, mineral water, tanning, electroplating, shampoo preparations, process engraving and lithography, soft soaps, textile dyeing, bleaching and finishing oil, and photography.

The composition of committee responsible for the formulation of this standard is given at Annex C.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

# Indian Standard

## POTASSIUM CARBONATE, ANHYDROUS — SPECIFICATION

( Second Revision )

### 1 SCOPE

This standard prescribes the requirements and methods of sampling and test for potassium carbonate, anhydrous.

### 2 REFERENCES

The Indian Standards given below contain provisions which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

<i>IS No.</i>	<i>Title</i>
323 : 2009	Rectified spirit ( <i>second revision</i> )
1070 : 1992	Reagent grade water ( <i>third revision</i> )
3025 (Part 2) : 2019/ISO 11885 : 2007	Methods of sampling and test (physical and chemical) for water and wastewater: Part 2 Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) ( <i>first revision</i> )
4161 : 1967	Nessler cylinders
4905 : 2015	Random sampling and randomization procedures ( <i>first revision</i> )
7224 : 2006	Iodized salt, vacuum evaporated iodized salt and refined iodized salt — Specification ( <i>second revision</i> )

### 3 GRADES

The material shall be of two grades, namely:

- a) Pure grade, and
- b) Technical grade.

### 4 REQUIREMENTS

#### 4.1 Description

The material shall be a white deliquescent crystalline powder or granule.

**4.2** The particle size for the granular form shall be as agreed to between the purchaser and the supplier.

**4.3** The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the Table.

#### 4.4 Optional Requirement

The material when intended to be used as dehydrating agent shall be dried at 120 °C.

### 5 PACKING AND MARKING

#### 5.1 Packing

The material shall be packed in clean, dry HDPE or PP bags with HM-HDPE liner inside. The liner should be airtight knot after filling the material, bag should be properly stitched with double stitch or the material shall be packed in clean, dry and airtight drum of appropriate MOC or as agreed to between the purchaser and the supplier. The containers shall not be opened until required for use and shall not remain open for a period longer than required for taking out material.

#### 5.2 Marking

**5.2.1** Each container shall bear legibly and indelibly the following information:

- a) Name and grade of the material,
- b) Indication of the source of manufacture,
- c) Mass of the material in the container,
- d) Date of packing, and
- e) Batch number.

#### 5.2.2 BIS Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

### 6 SAMPLING

The procedure for drawing representative samples of the material and their criteria for conformity shall be as prescribed in Annex B.

**Table 1 Requirements for Potassium Carbonate, Anhydrous**  
( Clause 4.3 )

Sl No.	Characteristic	Requirement (On Received Basis)		Method of Test (Ref to Cl No. in Annex A)
		Pure Grade	Technical Grade	
(1)	(2)	(3)	(4)	(5)
i)	Total alkalinity (as $K_2CO_3$ ), percent by mass, <i>Min</i>	99.0	99.0	A-2
ii)	Potassium bicarbonate (as $KHCO_3$ ), percent by mass, <i>Max</i>	0.4	0.4	A-3
	<i>OR</i>			
	Free alkali (as KOH), percent by mass, <i>Max</i>	0.5	0.5	
iii)	Sulphates (as $K_2SO_4$ ), percent by mass, <i>Max</i>	0.05	0.2	A-4
iv)	Chlorides (as KCl), percent by mass, <i>Max</i>	0.015	0.1	A-5
v)	Matter insoluble in water, percent by mass, <i>Max</i>	0.1	0.2	A-6
vi)	Iron (as Fe), percent by mass, <i>Max</i>	0.002	0.005	A-7 & A-12
vii)	Heavy metals (as Pb), percent by mass, <i>Max</i>	0.001	—	A-8 & A-12
viii)	Loss on heating at 150 °C, percent by mass, <i>Max</i>	1.0	1.0	A-9
ix)	Sodium (as Na), percent by mass, <i>Max</i>	1.0	2.0	A-10 & A-12
x)	Calcium, percent by mass, <i>Max</i>	0.002	0.002	A-11 & A-12
xi)	Magnesium, percent by mass, <i>Max</i>	0.001	0.001	A-11 & A-12

## ANNEX A

[ Clause 4.3, Table 1 ]

## METHODS OF TEST FOR POTASSIUM CARBONATE, ANHYDROUS

## A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 TOTAL ALKALINITY (EXPRESSED AS  $K_2CO_3$ )

## A-2.1 Reagents

A-2.1.1 *Standard Hydrochloric Acid* — 1 N.

A-2.1.2 *Methyl Orange Indicator Solution* — Dissolve 0.1 g of methyl orange in 100 ml of water.

## A-2.2 Procedure

Weigh quickly about 20 g of the sample (accurate to 0.01 g) in a stoppered weighing bottle and dissolve in about 100 ml of water. Filter through a sintered crucible No 4 and transfer into a 250 ml volumetric flask. Wash thoroughly with water and add the washings to the filtrate. Dilute the resulting solution to 250 ml mark and shake well. Now take 25 ml of the aliquot and titrate it against standard 1 N HCl towards the end point using methyl orange indicator.

## A-2.3 Calculation

Total alkalinity (as  $K_2CO_3$ ), percent by mass

$$= \frac{69.1 V N}{M}$$

where

V = Volume in ml, of standard 1 N HCl,

N = Actual normality of standard 1 N HCl, and

M = Mass in g, of the material in the sample solution taken for the test (*see* A-2.2)

## A-3 POTASSIUM BICARBONATE AND FREE ALKALI

## A-3.1 Reagents

A-3.1.1 *Barium Chloride Solution* — Approximately 1 N, neutral to phenolphthalein.

A-3.1.2 *Sodium Hydroxide Solution* — 0.1 N, free from carbon dioxide.

A-3.1.3 *Standard Hydrochloric Acid* — 0.1 N.

A-3.1.4 *Phenolphthalein Indicator*

Dissolve 0.1 g of phenolphthalein in 60 ml of rectified spirit (conforming to IS 323), and dilute with water to 100 ml.

## A-3.2 Procedure

Weigh quickly about 20 g of the sample (accurate to 0.01 g) in a stoppered weighing bottle and dissolve in about 100 ml of water. Filter through a sintered crucible No 4 and transfer into a 250 ml volumetric flask. Wash thoroughly with water and add the washings to the filtrate. Dilute the resulting solution to 250 ml mark and shake well. Transfer 25 ml of the sample solution into a conical flask. Add 10 ml of sodium hydroxide solution followed by 170 ml of barium chloride solution which is added slowly with constant shaking. Add 0.2 ml (4 drops) of phenolphthalein indicator, stopper the flask, mix the contents thoroughly and let it stand for five minutes. Titrate with the standard hydrochloric acid until the pink colour is discharged.

## A-3.3 Calculation

a) Potassium bicarbonate as  $(KHCO_3)$ ,

$$\text{percent by mass} = \frac{10(10 - V)}{M}$$

when  $V$  is less than 10 ml of NaOH added.

b) Free alkali (as KOH), percent by mass

$$= \frac{5.6(V - 10)}{M}$$

when  $V$  is greater than 10 ml of NaOH added

where

$V$  = volume in ml, of standard hydrochloric acid used, and

$M$  = mass in g, of the material taken for the test.

## A-4 DETERMINATION OF SULPHATES

## A-4.1 Reagents

A-4.1.1 *Dilute Hydrochloric Acid*, Approximately 10 percent ( $v/v$ ).

A-4.1.2 *Barium Chloride Solution*, Approximately 10 percent ( $m/v$ ).

## A-4.2 Procedure

Weigh accurately about 5 g of the material and add 30 ml of dilute hydrochloric acid. Filter, if necessary, and wash the filter paper thoroughly. Heat the solution to boiling and add drop by drop 10 ml of hot barium chloride solution. Boil the solution for 15 min and then allow to stand for 4 h. Filter through a tared Gooch or a sintered glass crucible (G No. 4). Wash the precipitated barium sulphate with hot water till it is free from chlorides and dry to constant mass at about 110 °C.

**A-4.3 Calculation**

Sulphates as ( $K_2SO_4$ ), percent by mass

$$= 74.63 \times \frac{M_1}{M}$$

where

$M_1$  = mass of barium sulphate, and

$M$  = mass of the material taken for the test.

**A-5 TEST FOR CHLORIDES**

**A-5.1** Two methods are prescribed, one is Nessler cylinder method and the other is volumetric method. In case of dispute Nessler cylinder method shall be the referee method.

**A-5.2 Nessler Cylinder Method****A-5.2.1 Apparatus**

**A-5.2.1.1** Nessler cylinders — 50-ml capacity.

**A-5.2.2 Reagents**

**A-5.2.2.1** Dilute nitric acid—Approximately 5 N.

**A-5.2.2.2** Silver nitric solution — Approximately 5 percent.

**A-5.2.2.2** Silver nitrate solution — Approximately 5 percent.

**A-5.2.2.3 Standard chloride solution**

Dissolve 0.165 g of sodium chloride, dried at  $105 \pm 2^\circ C$  in water and make up to 1 000 ml. One millilitre of this solution contains 0.1 mg of chloride (as Cl).

**A-5.2.3 Procedure**

Weigh 10.0 g for pure grade or 1.0 g for technical grade of the material into a Nessler cylinder, dissolve in 30 ml of water and add 15 ml of dilute nitric acid followed by 1 ml of silver nitrate solution and dilute to the mark. Carry out a control test in another Nessler cylinder using 7 ml of standard chloride solution for pure grade and 5 ml for technical grade in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture and compare the opalescence produced in two cylinders after two minutes.

**A-5.2.3.1** The limit prescribed for chloride shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than that produced in the control test.

**A-5.3 Volumetric Method****A-5.3.1 Reagents**

**A-5.3.1.1** Silver nitrate solution (0.01 N).

Weigh accurately 1.699 g of silver nitrate (AR grade) and dissolve in chloride free water. Add 10 ml of concentrated nitric acid and dilute to 1 000 ml with water. Transfer the solution to a coloured bottle and store in dark place.

**A-5.3.1.2** Potassium thiocyanate solution (0.01N).

Weigh accurately 0.971 7 g of potassium thiocyanate (AR grade), dissolve in water and dilute to 1 000 ml. Standardize this solution with standard 0.01 N silver nitrate solution.

**A-5.3.1.3** Ferric nitrate solution (40 percent)

Weigh 40 g of ferric nitrate (AR grade) and dissolve in a small quantity of water. Add a few drops of concentrated nitric acid (AR grade) and dilute to 100 ml.

**A-5.3.1.4** Potassium chloride solution (0.02 percent)

Weigh accurately 0.2 g of potassium chloride (AR grade), dissolve in chloride free water and dilute to 1 000 ml.

**A-5.3.1.5** Concentrated nitric acid

**A-5.3.1.6** Nitrobenzene

**A-5.3.2 Procedure**

**A-5.3.2.1** Take 10 ml of standard potassium chloride solution (**A-5.3.1.4**) and add about 50 ml of chloride free water. Add 5 ml of concentrated nitric acid and 2 ml of ferric nitrate solution (**A-5.3.1.3**). Add 10 ml of 0.01 N silver nitrate solution (**A-5.3.1.1**) followed by 2 ml of nitrobenzene, stir vigorously and titrate with 0.01 N potassium thiocyanate solution till the faint brick red colour appears. From the consumption of standard silver nitrate solution, calculate the equivalent of potassium chloride content for 1 ml of silver nitrate solution.

Let 1 ml of standard silver nitrate solution =  $X$  mg of potassium chloride.

**A-5.3.2.2** Weigh 10 g of potassium carbonate sample and transfer to a 250-ml conical flask. Add a few millilitres of water and neutralize with concentrated nitric acid. Add 2 ml of ferric nitrate indicator (**A-5.3.1.3**) and 10 ml of 0.01 N standard silver nitrate solution. Add 2 ml of nitrobenzene and titrate against 0.01 N potassium thiocyanate solution (**A-5.3.1.2**) till brick red colour appears.

**A-5.3.3 Calculation**

Chloride (as KCl), percent by mass

$$= \frac{(10 - V) \times X}{10 M}$$

where

$V$  = volume in ml, of standard potassium thiocyanate solution used in the titration,

$X$  = mass in mg, of potassium chloride equivalent to 1 ml of silver nitrate, and

$M$  = mass in g, of the material taken for the test.

**A-6 DETERMINATION OF MATTER INSOLUBLE IN WATER****A-6.1 Procedure**

Weigh accurately 5 to 10 g of the material, transfer it to a 400-ml beaker, add about 200 ml of freshly boiled water, and boil the resulting solution for about 10 min. Filter through a tared Gooch crucible or a sintered glass crucible (G. No. 4) and wash the residue with hot water till it is free from soluble salts. Dry the crucible along with the insoluble residue at 110 °C and weigh to constant mass.

**A-6.2 Calculation**

Matter insoluble in water, percent by mass =  $\frac{M_1}{M} \times 100$   
where

$M_1$  = mass in g, of the residue, and

$M$  = mass in g of the material taken for the test.

**A-7 TEST FOR IRON**

General — Two methods are prescribed. In case of dispute, the spectrophotometric method shall be used.

**A-7.1 Method A****A-7.1.1 Apparatus**

**A-7.1.1.1 Nessler cylinder** — 50-ml capacity (see IS 4161).

**A-7.1.2 Reagents**

**A-7.1.2.1 Dilute hydrochloric acid** — Approximately 5 N.

**A-7.1.2.2 Ammonium persulphate**

**A-7.1.2.3 Potassium thiocyanate Solution** — Approximately 5 percent.

**A-7.1.2.4 Dilute sulphuric acid** — 10 percent (v/v).

**A-7.1.2.5 Standard iron solution**

Dissolve 0.702 g of ferrous ammonium sulphate in 10 ml of dilute sulphuric acid and dilute with water

to 1 000 ml. Dilute 100 ml of this solution again to 1 000 ml. One millilitre of the diluted solution contains 0.01 mg of iron (as Fe).

**A-7.1.3 Procedure**

Weigh accurately, 1000 g of the material and dissolve in about 20 ml of water. Add about 5 ml of hydrochloric acid and 30 mg of ammonium persulphate, and boil to oxidize the iron. Cool and transfer to a Nessler cylinder, add 5 ml of potassium thiocyanate solution, dilute with water to 50 ml mark and stir well. Carry out a control test using 2 ml of the standard iron solution for pure grade and 5 ml for technical grade in place of the material and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in the cylinders after 5 min.

**A-7.1.3.1** The limit prescribed in Table 1 shall be taken as not having been exceeded if the intensity of colour produced in the test with the material is not greater than that produced in the control test.

**A-7.2 Method B (Spectrophotometric Method)**

**A-7.2.1 General** — Ferrous iron reacts with *o*-phenanthroline between pH 3 and 9 in the ratio 1:3 to give a stable, orange-red coloured complex which has maximum absorbance at 510 nm.

**A-7.2.2 Apparatus** — Spectrophotometer.

**A-7.2.3 Reagents**

**A-7.2.3.1 Standard iron solution** — Dissolve 7.02 g of ferrous ammonium sulfate  $\{\text{FeSO}_4 (\text{NH}_4)_2 \text{SO}_4 \cdot 6\text{H}_2\text{O}\}$  in water containing 10 ml of concentrated sulphuric acid and dilute with water to 1 000 ml. One millilitre of this solution contains 1.0 mg of iron (as Fe)}. It may be diluted suitably to contain 10, 25 or 100 µg of iron per millilitre.

**A-7.2.3.2 Hydroxylamine hydrochloride solution** — Dissolve 10 g in water and dilute to 100 ml. This solution should be freshly prepared.

**A-7.2.3.4 *o*-phenanthroline solution** — Dissolve 0.1 g of *o*-phenanthroline in 50 ml of ethyl alcohol and dilute to 100 ml with water. Mix well and store in a dark bottle with a ground-glass stopper.

**A-7.2.3.5 Sodium acetate-acetic acid buffer solution** — Dissolve 28 g of glacial acetic acid in water and dilute to 500 ml. Similarly dissolve 68 g of sodium acetate in 500 ml of water. At the time of use, prepare the required quantity of buffer solution by mixing equal volumes of the two solutions.



**A-7.2.3.6 Dilute sulphuric acid** — approximately 4 N.

**A-7.2.3.7 Dilute ammonium hydroxide**

**A-7.2.4 Preparation of Standard Graph**

Take 5 ml of standard iron solution (10 µg/ml) into a 100 ml beaker. Add 10 ml of water, 1 ml of hydroxylamine hydrochloride solution, 1 ml of dilute sulphuric acid and 5 ml of orthophenanthroline solution. Add sufficient ammonia solution to adjust the pH of the solution to 5 and then add 5 ml of buffer solution. Adjust the pH to 5 again (if necessary). Transfer the contents to a 100 ml volumetric flask and dilute to the mark with water. Keep aside the flask for 30 min to develop red colour fully. This solution corresponds to iron concentration of 0.05 mg/100 ml. Determine the optical density of this solution on the spectrophotometer at 510 nm. Similarly prepare solutions of iron concentrations 0.10 mg, 0.15 mg, 0.20 mg, 0.25 mg and 0.30 mg per 100 ml. Determine optical densities of these solutions and plot a graph of concentrations against corresponding optical densities.

**A-7.2.5 Procedure**

Weigh accurately 10 g of the material and transfer into a one-litre conical flask. Add 150 ml of water and sufficient hydrochloric acid to dissolve the sample. When effervescence ceases, boil the contents to evolve out the carbon dioxide. Cool to room temperature and dilute to 250 ml in a volumetric flask. Take 25 ml of the solution in a 100 ml beaker, add 1 ml of hydroxylamine hydrochloride solution and 5 ml of orthophenanthroline solution. Adjust the pH of the solution to 5 and then add 5 ml of buffer solution. Adjust the pH to 5 again (if necessary) and dilute to the mark with water. Keep the contents for 30 minutes to develop the red colour fully and then determine the optical density on the spectrophotometer at 510 nm. From the standard graph, read the corresponding concentration of iron per 100 ml of the solution.

**A-7.2.6 Calculation**

$$\text{Iron content, percent by mass} = \frac{c \times d \times 100}{M}$$

where

$c$  = concentration of iron in mg, per 100 ml of the sample solution,

$d$  = dilution factor, and

$M$  = mass in mg, of the sample taken for analysis.

NOTE —If the sample solution is strongly red coloured suitably less volumes, namely, 5 ml or 10 ml may be taken for analysis.

**A-7.3** Alternatively, Iron can also be determined by ICP-OES method as given in **A-12**.

## **A-8 TEST FOR HEAVY METALS (as Pb)**

### **A-8.1 Method A**

#### **A-8.1.1 Apparatus**

**A-8.1.1.1 Nessler cylinders** — 50-ml capacity (see IS 4161).

#### **A-8.1.2 Reagents**

**A-8.1.2.1 Dilute hydrochloric acid** — Approximately 5 N.

**A-8.1.2.2 Dilute ammonium hydroxide solution** — Approximately 5 N.

#### **A-8.1.2.3 Potassium cyanide solution**

Dissolve 10 g of potassium cyanide in 100 ml of water.

#### **A-8.1.2.4 Acetic acid** — Glacial.

#### **A-8.1.2.5 Hydrogen sulphide solution**

Saturated and freshly prepared hydrogen sulphide in water.

#### **A-8.1.2.6 Standard lead solution**

Dissolve 1.60 g of lead nitrate in water and make up the solution to 1 000 ml with water. Dilute 10 ml of this solution to 1000 ml with water. One millilitre of the diluted solution contains 0.01 mg of lead (as Pb).

#### **A-8.1.3 Procedure**

Weigh 1.000 g of the material and dissolve in 20 ml of water in a Nessler cylinder. Add 5 ml of dilute hydrochloric acid, 10 ml of dilute ammonium hydroxide solution, 1 ml of potassium cyanide solution and dilute to 40 ml. Add 5 ml of hydrogen sulphide solution, dilute to the mark and mix well. Carry out a control test using 1 ml of standard lead solution, 1 ml of acetic acid and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour produced in both the cylinders after 5 min.

**A-8.1.3.1** The limit prescribed for heavy metals (as Pb) shall be taken as not having been exceeded if any colour produced in the test with the material is not darker than that produced in the control test.

### **A-8.2 Method B (Spectrophotometric Method)**

#### **A-8.2.1 Principle**

An sample containing microgram quantities of lead is extracted with dithizone solution in chloroform. The extraction is carried out in the presence of strong ammoniacal citrate-cyanide reducing agent (pH 10 to 11.5). The quantity of lead present in the



sample is determined spectrophotometrically by measuring the absorbance at 510 nm of the chloroform extract containing the lead dithizonate complex.

#### **A-8.2.2 Minimum Detection Limit**

1.0 µg Pb/10 ml dithizone solution (extract).

#### **A-8.2.3 Apparatus**

Spectrophotometer for use at 510 nm with a path length of 1 cm or longer.

*pH meter*

*Standard Volumetric Glasswares*

*TEF Beaker*, 100 ml for Acid Digestion.

*Separatory Funnels*, 250 ml and 500 ml.

All glasswares are to be cleaned with 1 : 1 HNO<sub>3</sub>, and rinsed thoroughly with distilled water.

#### **A-8.2.4 Reagents**

##### **A-8.2.4.1 Quality of reagents**

Only analytical or equivalent grade reagents, unless specified otherwise, are to be used. All reagents are to be prepared in lead-free distilled water.

##### **A-8.2.4.2 Stock lead solution**

Dissolve 0.1599 g lead nitrate [(Pb(NO<sub>3</sub>)<sub>2</sub>), minimum purity, 99.5 percent (w/w)] in about 200 ml of water. Add 10 ml concentrated HNO<sub>3</sub> and dilute to 1000 ml with water, 1.0 ml of this solution will contain 100 µg of Pb.

##### **A-8.2.4.3 Standard lead solution**

Dilute 2.0 ml of stock lead solution to 100 ml with water, 1.0 ml of this solution will contain 2 µg of Pb.

##### **A-8.2.4.4 Nitric acid — Concentrated (18N).**

##### **A-8.2.4.5 Nitric acid — Dilute — 20 percent, v/v.**

##### **A-8.2.4.6 Ammonium hydroxide — Concentrated (14N).**

##### **A-8.2.4.7 Ammonium hydroxide — Dilute 10 percent, v/v and 1 percent, v/v.**

**A-8.2.4.8 Citrate-cyanide reducing Solution —** Dissolve 200 g anhydrous ammonium citrate [(NH<sub>4</sub>)<sub>2</sub> HC<sub>6</sub>H<sub>5</sub>O<sub>7</sub>], 10 g anhydrous sodium sulphite (Na<sub>2</sub>SO<sub>3</sub>), 5 g hydroxylamine hydrochloride, 20 g potassium cyanide (KCN) in water and dilute to 500 ml, and mix with one litre of concentrated NH<sub>4</sub>OH.

CAUTION — KCN is a poisonous solution. Handle with extreme care and do not pipette by mouth.

##### **A-8.2.4.9 Stock dithizone solution**

Dissolve 25 mg dithizone in about 50 ml chloroform (CHCl<sub>3</sub>) taken in a 200 ml beaker and filter through Whatman No. 42 (or equivalent) filter paper. Collect

the filtrate and two washings (10 ml each) in a 250 ml conical flask. Transfer the combined filtrate to a 500 ml separatory funnel. Add about 100 ml 1 percent (v/v) NH<sub>4</sub>OH solution, shake moderately for about 1 min. Transfer the CHCl<sub>3</sub> layer to another 250 ml separatory funnel retaining the orange-red aqueous layer in the 500 ml separatory funnel. Repeat the extraction (of the CHCl<sub>3</sub> layer) with 100 ml of 1 percent (v/v) NH<sub>4</sub>OH solution, transfer the CHCl<sub>3</sub> layer to another 250 ml separatory funnel and the aqueous layer to the original 500 ml separatory funnel containing the first extract. One more repetition, of extraction and transferring to the main aqueous layer is carried out. To the combined aqueous extract in the 500 ml separatory funnel add 1 : 1 HCl in 2 ml portions, mixing after each addition, until dithizone precipitation is complete and the solution is no longer orange-red. Extract the precipitated dithizone with three 25 ml portions of CHCl<sub>3</sub>. Dilute the combined extract to 250 ml with CHCl<sub>3</sub>, 1 ml of this solution will contain 100 µg of dithizone.

##### **A-8.2.4.10 Working dithizone solution**

Dilute 100 ml stock dithizone solution to 250 ml in a standard volumetric flask with CHCl<sub>3</sub>, 1 ml of this solution will contain 40 µg of dithizone.

#### **A-8.2.5 Procedure**

##### **A-8.2.5.1 Sample digestion**

Digest all samples for lead as per standard digestion procedure using HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub> and HNO<sub>3</sub>-HClO<sub>4</sub>.

To 100 ml acidified sample (pH = 2) add 20 ml of dilute (20 percent, v/v) HNO<sub>3</sub>, filter if required through a filter paper (Whatman No. 41 or equivalent), and transfer it to a 250 ml separatory funnel. Add 60 ml ammoniacal citrate-cyanide solution, mix and cool to room temperature. Add 10 ml of dithizone working solution. Shake the stoppered funnel vigorously for about 30s, allow to stand (to get two separate layers). Discard 1-2 ml CHCl<sub>3</sub> layer and then fill the absorption cell. Measure the absorbance at 510 nm using working dithizone solution as reagent blank.

##### **A-8.2.5.2 Calibration curve**

Plot a calibration curve using at least five standard lead solutions, after adding 50 ml ammoniacal citrate-cyanide solution to the individual lead standard solutions and extracting the same with 10 ml of dithizone working solution.

##### **A-8.2.5.3 Calculation**

**mg Pb/litre** = µg (in 10 ml extract obtained from calibration curve)/volume of sample (ml).

**A-8.3** Lead can also be analysed by ICP-OES method as given in **A-12**.

**A-8.4** Alternatively, lead can also be determined as per Atomic Absorption Spectrophotometer (AAS) method as prescribed in IS 7224.

## **A-9 DETERMINATION OF LOSS ON HEATING AT 150 °C**

### **A-9.1 Apparatus**

#### **A-9.1.1 Weighing Bottle**

Approximately 100 ml capacity and 75 mm in diameter, with ground glass stopper.

#### **A-9.1.2 Electric Oven**

Capable of reaching a temperature of at least 150 °C and regulated so as not to exceed a temperature of 170 °C.

### **A-9.2 Procedure**

Weigh the weighing bottle previously dried for 30 min in the oven controlled at a temperature of 150 °C and allowed to cool to room temperature in a desiccator. Spread thin layers of the test portion in the weighing bottle and weigh to the nearest milligram  $5 \pm 0.1$  g of the sample. Place the weighing bottle with the ground glass stopper slantwise in the oven set at a temperature of 100 °C. Gradually heat the sample in the oven from the starting temperature (which should not exceed 100 °C) to at least  $150 \pm 2$  °C and maintain this temperature for 4 h. Remove the weighing bottle, place it in a desiccator to cool, and after cooling to room temperature, weigh it again (with stopper in position) to constant mass.

### **A-9.3 Calculation**

Loss on heating at 150 °C, percent by mass =

$$\frac{(M_1 - M_2) \times 100}{M}$$

where,

$M_1$  = mass in g, of the material and weighing bottle before heating,

$M_2$  = mass in g, of the material and weighing bottle after heating, and

$M$  = mass in g, of the material taken for the test.

## **A-10 DETERMINATION OF SODIUM**

### **A-10.1 Method A**

#### **A-10.1.1 Apparatus**

**A-10.1.1.1 Flame photometer** — equipped with interference filter.

NOTE — Sodium filter to be used should be of preselected or metal filter type, since ordinary sodium filters (for example, gelatine fillers) have only 1 percent selectivity and work properly when the ratio of Na:K is 100:1, whereas in potassium carbonate and potassium hydroxide, the ratio is exactly reverse. Hence results with gelatine filter are erratic.

### **A-10.1.2 Reagent**

#### **A-10.1.2.1 Standard sodium solution**

Weigh exactly 2.542 g of sodium chloride, dissolve in deionized water and dilute to 1 000 ml in a measuring flask. Dilute 10 ml of this solution to 1000 ml. 1ml of the diluted solution contains 0.01 mg of sodium (as Na).

#### **A-10.1.3 Sample Solution**

Dissolve exactly 10 g of the pure grade of material (5 g in case of technical grade) in deionized water and dilute to 1 000 ml in a volumetric flask. Dilute 100 ml of this solution to 1 000 ml in a volumetric flask. Use this solution for galvanometer readings. If needed, dilute the solution further.

#### **A-10.1.4 Calibration Graph**

Take 10, 20, 40, 60 and 80 ml of standard sodium solution in different measuring flasks and dilute to 100 ml with deionized water. Each flask now contains 0.1, 0.2, 0.4, 0.6 and 0.8 mg of sodium per 100 ml. Use these diluted solutions to obtain corresponding galvanometer reading as given in the procedure (A-10.1.5.2) and plot the concentration against galvanometer reading in a rectangular coordination graph. Draw a smooth curve over the points which gives a calibration graph in the range 0.1 to 1.0 mg of sodium per 100 ml.

#### **A-10.1.5 Procedure**

**A-10.1.5.1** Use a flame photometer equipped with atomizer, burner, optical selective device consisting of reflectors, lenses and diaphragms; and measuring instrument consisting of a photocell, an amplifier and a sensitive galvanometer. The galvanometer scale ranges from 0 to 100 divisions which measures the intensity of the radiation transmitted by the element.

**A-10.1.5.2** Insert the sodium filter corresponding to wavelength 767 nm. Light the burner fed by illuminating gas (laboratory gas) and adjust the specified air pressure between 0.5 to 0.7 kg/cm<sup>2</sup> and maintain the control knob. First spray deionised water and adjust the pointer to zero in galvanometer scale by adjustment knob. Then spray the standard sodium solution and adjust the deflection to maximum (100) by using sensitivity control knob. Again spray deionised water to see that pointer comes to zero; then spray standard solution to indicate 100. Repeat till deionized water reads zero and standard solution reads 100 with the same adjustment during both the operations. Reading zero by deionized water and with the same adjustment 100 by standard solution, indicates that the instrument is now ready for measurement.

**A-10.1.5.3** Without altering the earlier adjustment of the instrument, spray various diluted solutions prepared

in **A-10.4** and obtain a calibration graph in the range of 0.1 to 1.0 mg sodium per 100 ml. After washing with deionised water, spray the sample solution and obtain the galvanometer reading. From the graph, read out the corresponding concentration of sodium (say A).

#### **A-10.1.6 Calculation**

Sodium (as Na), percent by mass =  $10 \frac{A}{M}$

where

A = concentration of sodium in mg, per 100 ml corresponding to the galvanometer reading, and

M = mass in g, of the material taken for preparing the sample solution.

**A-10.2** Alternatively, Sodium can also be determined by ICP-OES method as given in **A-12**.

### **A-11 DETERMINATION OF CALCIUM AND MAGNESIUM**

#### **A-11.1 Calcium Determination**

Take 50 ml of test solution (concentration of 100g of sample/1000 ml) in a 250 ml conical flask. Add 2 ml triethanolamine, 10 ml sodium hydroxide, 15 ml ethanol and 1 ml glyoxal-bis-(2-hydroxyanil) solution. The solution should be highly basic with a pH of around 12. Adjust the pH by adding sodium hydroxide. Keep the solution for about 2 min and then titrate against EDTA till the colour of the solution changes from red to yellow. The titration time should be less than 3 min.

#### **A-11.2 Total determination of Calcium and Magnesium**

Take 100 ml of solution (100g in 1000 ml) and add it in a 500 ml conical flask. Dilute the mixture to 200 ml and add 2 ml of triethanolamine solution. Further add 5 ml of buffer solution and EDTA in amount slightly less than twice the volume of EDTA used for calcium determination. Heat the mixture to around 50 degree Celsius and add 0.1 ml of mordant black 11. Continue to add EDTA solution till the end point changes from wine red to blue.

#### **A-11.3 Results**

##### **A-11.3.1 Calcium Content**

Calcium soluble in water is given as:

$$V \times 0.02 \times 0.04008 \times (1000/50) \times (100/m)$$

where V is the volume (in ml) of EDTA which is used for determination of calcium.

M is mass of test sample

$0.02 \times 0.04008$  is the calcium (in grams) corresponding to 1 ml of EDTA solution.

##### **A-11.3.2 Magnesium Content**

Amount of magnesium (percentage by mass) is given as:

$$(V_1 - 2V) \times 0.02 \times 0.02432 \times (1000/100) \times (100/m)$$

Where  $V_1$  is the volume in ml of EDTA which is required for the determination of calcium and magnesium.

**A-11.4** Alternatively, Calcium and Magnesium can also be determined by ICP-OES method as given in **A-12**.

### **A-12 DETERMINATION OF LEAD, IRON, CALCIUM, MAGNESIUM, SODIUM BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETER (ICP-OES) METHOD**

#### **A-12.1 Principle**

The sample solution under analysis is nebulized through a nebulizer inside a spray chamber. The aerosol formed is aspirated to argon plasma torch [produced by a radio-frequency inductively Coupled Plasma (ICP)], where the molecules break into constituent atoms and/or molecular species and atoms are get excited. These excited atoms then return back to the lower energy state by emitting radiation of specific wavelength. These emitted radiations are characteristic of an element and are measured by the Photomultiplier tube detector and intensity of such emitted radiation is directly proportional to the concentration of respective constituent element in the sample.

#### **A-12.2 Recommended Wavelength, Limit of Quantification and Important Spectral Interferences**

Elements along with the recommended wavelengths and typical estimated limits of quantification are listed in Table 2. Actual working detection limits are dependent on the type of instrumentation, detection device and sample introduction system used and on the sample matrix. Therefore, these concentrations can vary between different instruments.

Additionally, Table 1 lists the most important spectral interferences at the recommended wavelengths for analysis.

**Table 2 Recommended Wavelengths, Achievable Limits of Quantification for Different Configuration of Instruments and Important Spectral Interferences**

Sl No.	Element	Wavelength (nm)	Approx. Achievable limits		Interfering Elements
			Radial viewing (µg)	Axial viewing (µg)	
(1)	(2)	(3)	(4)	(5)	(6)
i)	Pb	220.353	14	5	Al, Co, Fe, Ti
		283.305	(70)	(20)	Cr, Fe
ii)	Fe	238.204	14	(3)	Co
		259.940	6	2	Co
		271.441	—	—	—
iii)	Ca	315.887	100	13	Co, Mo
		317.933	26	4	Fe, V
		393.366	0.4	25	V, Zr
		422.673	—	—	V, Mo, Zr
iv)	Mg	279.078	33	19	Fe
		279.553	1	7	Fe
		285.213	4	14	Cr
v)	Na	330.237	20	300	Zn
		588.995	20	200	Ar, V
		589.592	93	20	Ba

**A-12.3 Reagents and Solutions****A-12.3.1 Nitric acid (65 Percent) Suprapure**

**A-12.3.2 Standard Stock Solution** — Either prepare by dissolving proportionate amount of soluble compounds of elements (preferably spectroscopic grade), or use commercially available certified stock solution of 10, 100 or 1 000 µg/ml of lead, Iron, calcium, magnesium, manganese, sodium in 2-5 percent nitric acid. It is preferable to prepare single stock solution of multi elemental standards for analysis.

**A-12.3.3 Standard Solution** — Pipette out 5 ml from 100 µg/ml standard stock solution into a 100 ml volumetric flask and make up volume with 2 percent nitric acid to prepare 5 µg/ml solution. From this 5 µg/ml solution, an aliquot of 1.0, 3.0 and 5.0 ml taken in 50 ml volumetric flasks (separate) and make up volume with 2 percent nitric acid to prepare 0.1, 0.3 and 0.5 µg/ml solution of respective elements under reference.

**A-12.3.4 Sample Preparation** — Weigh about 2.5 g polyphosphoric acid sample in a 50 ml volumetric flask and add 1.0 ml Nitric acid and make up the volume with water.

**A-12.3.5 Reagent Blank Solution** — Place 50 ml of nitric acid and 1 000 ml of water into an HDPE or PP container. For ultra-trace analysis, polytetrafluorethylene (PTFE) containers should be used. Prior to analysis, make sure that the acid matrix

and concentration of the reagent blank solution is the same as in the standard and sample solutions.

**A-12.4 Instrument**

Set up the instrument as per the manufacturer's instructions manual for recommended operating parameters, based on the manufacturers operating manual and evaluated by internal check analysis using of standard solution of element as well as data selected carefully from Table 2.

NOTE — Sensitivity, instrumental detection limit, precision, linear dynamic range and interference effects will be investigated and established for each individual analyte line on that particular instrument.

**A-12.5 Procedure****A-12.5.1 Calibration**

Profile and calibrate the instrument according to the instrument manufacturer's recommended procedures, using the intermediate mixed standard solutions (A-12.3.3). The relationship between concentration and intensity is linear up to six orders of magnitude. Examine the spectra of the element and make necessary adjustments (if required) for the exact peak positions and baselines to ensure proper measurements of the respective peak intensities. Flush the system with the reagent blank solution between each standard.

**A-12.5.2** Before beginning the sample run, re-analyse the reference standard with the highest concentration as if it were a sample. Ensure that the concentration values do not deviate from the actual values by more than

± 5 percent (or the established control limits, whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

Begin the sample run by flushing the system with the reagent blank solution (A-12.3.5) between each sample. It is recommended to analyse a calibration check solution and the calibration blank solution every 10 samples.

Analyze the sample solution and calculate the concentration in µg/ml of the lead (and/or Iron, calcium, magnesium, sodium) in the sample solution.

NOTE — It is recommended that IS 3025(Part 2)/ISO 11885 may be referred and practiced for ensuring precise and reproducible analysis.

## A-12.6 Calculation

The mass concentrations for each element are determined with the aid of the instrument software by following steps.

- Relate emission signals from calibration blank and calibration solutions with the signals from reference elements and establish a calibration plot.
- Determine the mass concentrations of samples with the aid of the emissions and the calibration graphs and calculate the quantity in mg/kg of the constituent elemental impurities in the sample, by multiplying the value by 20 (dilution factor).

## ANNEX B

( Clause 6 )

### METHODS OF SAMPLING OF POTASSIUM CARBONATE

#### B-1 GENERAL REQUIREMENTS OF SAMPLING

**B-1.1** In drawing, storing, preparing and handling test samples, the following precautions shall be observed.

**B-1.2** Samples shall not be taken at an open place.

**B-1.3** Precaution shall be taken to protect the samples, the sampling instrument and the containers for samples from adventitious contamination.

**B-1.4** To draw a representative sample, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

**B-1.5** The sample shall be placed in suitable, clean, dry and air-tight glass containers.

**B-1.6** Each sample container shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling and year of manufacture.

#### B-2 SCALE OF SAMPLING

##### B-2.1 Lot

All the packages in a single consignment of potassium carbonate drawn from a single batch of manufacture shall constitute a lot. If the consignment is declared to consist of different batches, the batches shall be marked separately and the group of packages in each batch shall constitute separate lots.

**B-2.1.1** The number of packages (n) to be drawn at random from the lot shall depend upon the size of the lot (N) and shall be in accordance with Table 3.

**Table 3 Number of Packages to be drawn for Sampling**

( Clause B-2.1.1 )

Lot Size (N)	No. of Packages to be Drawn (n)
(1)	(2)
4 to 5	3
51 to 100	4
101 to 150	5
151 to 300	7
301 and above	10

NOTE — When the size of the lot is 3 or less, the number of packages to be drawn and the criterion for judging conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

**B-2.1.2** These packages shall be selected at random from the lot and in order to ensure the randomness of sampling, a random number table (see IS 4905) may be used.

**B-2.1.3** Samples shall be tested for each lot for ascertaining conformity of the material to the requirements of this specification.

#### B-3 PREPARATION OF TEST SAMPLES

**B-3.1** From each of the packages selected according to B-2.1.2, a portion of the material about 100 g, shall be drawn with the help of a suitable sampling instrument.

**B-3.2** Out of these portions, equal quantities of the material shall be taken and mixed thoroughly to form a composite sample of about 300 g. The composite



sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third to be used as a referee sample.

**B-3.3** The remaining portion of the material from each package shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the package shall be marked for the purchaser, another for the supplier and the third to be used as a referee sample.

**B-3.4** All the individual samples and the composite sample shall be transferred to separate sample containers. All the containers shall be sealed and labelled with full identification particulars.

**B-3.5** The referee test samples consisting of a composite sample and a set of individual samples shall bear the seals of both the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier, to be used in case of dispute between the two.

#### **B-4 NUMBER OF TESTS**

**B-4.1** Tests for total alkalinity shall be conducted on each of the individual samples.

**B-4.2** Tests for all other characteristics given under **4.3** shall be performed on the composite sample (*see B-3.2*).

#### **B-5 CRITERIA FOR CONFORMITY**

##### **B-5.1 For Individual Samples**

From the test results for total alkalinity (as potassium carbonate) the average ( $X$ ) and the range ( $R$ ) or  $R$  shall be computed.

NOTE — Range is defined as the difference between the maximum and minimum of the individual test results. When there are 10 values, they shall be formed into two groups of five each in the same order in which the individual samples are numbered.

For each group range shall be calculated and  $R$  is the average of these two  $R$ 's.

**B-5.1.1** The lot shall be considered satisfactory in respect of the requirement for total alkalinity, as potassium carbonate if  $(X - 0.4 R)$  or  $(X - 0.4R)$  is greater than or equal to 98.

##### **B-5.2 For Composite Samples**

The lot shall be considered to have passed in respect of the remaining characteristics tested on the composite test sample, if the test results satisfy the corresponding requirements given under **4**.

**B-5.3** The lot shall be considered as conforming to the specification if it satisfies all the criteria given in **B-5.1** and **B-5.2**.

## ANNEX C

( Foreword )

## COMMITTEE COMPOSITION

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